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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BAUXITE

PART 3 DETERMINATION OF ALUMINA

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF BAUXITE

PART 3 DETERMINATION OF ALUMINA

(First Revision)

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Indian Standard
METHODS OF CHEMICAL ANALYSIS OF BAUXITE
PART 3 DETERMINATION OF ALUMINA
(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 3) (First Revision) was adopted by the Indian Standards Institution on 24 December 1985, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 2000-1962* which covered determination of loss on ignition; total silica, non-reactive and reactive silica; alumina; ferric oxide by dichromate, permanganate and colorimetric methods; titania by reduction (volumetric) and colorimetric methods; phosphorus; calcium oxide; magnesium oxide and manganous oxide was first published in 1962. While reviewing this standard the Sectional Committee felt the need for the revision of this standard. On the basis of experience gained during these years the Committee decided to revise this standard into different parts covering determination of each element in separate part, which on publication would supersede that element given in IS : 2000-1962*. This part covers determination of alumina. The other parts are:

Part 1	Determination of loss on ignition
Part 2	Determination of silica (as SiO_2, 0·5 to 10·0 percent)
Part 4	Determination of ferric oxide
Part 5	Determination of titania (0·5 to 12·0 percent)
Part 6	Determination of vanadium ($V < 0·1$ percent)
Part 7	Determination of phosphorus pentoxide ($P_2O_5 < 0·5$ percent)
Part 8	Determination of manganous oxide ($MnO < 0·5$ percent)

The procedures for determination of CaO, and MgO in bauxite are at present under investigation and will be given in subsequent part.

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*Methods of chemical analysis of bauxite.

0.3 In this revision the complexometric method for determination of aluminium replaces the difference method given in the existing standard.

0.4 In preparation of this revision due consideration has been given to the facilities available in the country to carry out such analysis.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard covers the determination of alumina in bauxite.

2. SAMPLING

2.1 Sample shall be drawn and prepared as per the method prescribed in **2** of IS : 2000 (Part 1)-1985†.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

4. DETERMINATION OF ALUMINA

4.1 Complexometric Method

4.1.1 *Outline of the Method* — From an aliquot of the main solution, after dilution iron and titanium are precipitated by treatment with caustic soda solution. The precipitate is filtered through dry filter and alumina in the filtrate is determined complexometrically.

4.1.2 *Reagents*

4.1.2.1 *Caustic soda* — 20 percent (*m/v*).

4.1.2.2 *Concentrated hydrochloric acid* — rd = 1·16 (conforming to IS : 265-1976‡).

4.1.2.3 *Sodium carbonate* — solid.

4.1.2.4 *EDTA (0·05 M)* — Dissolve about 18·64 g of the disodium salt of EDTA $[(OOC.CH_2)_2NH.CH_2CH_2NH(CH_2COO)_2Na_2 2H_2O]$ in water and transfer to a 1 000-ml volumetric flask, dilute to the mark and mix.

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*Rules for rounding off numerical values (*revised*).

†Methods of chemical analysis of bauxite: Part 1 Determination of loss on ignition (*first revision*).

‡Specification for hydrochloric acid (*second revision*).

4.1.2.5 *Methyl red indicator solution* — 0·1 percent (*m/v*) in rectified spirit.

4.1.2.6 *Dilute hydrochloric acid* — 1:1 (*v/v*).

4.1.2.7 *Buffer solution* (*p*H 5·3) — Dissolve 21·5 g of sodium acetate ($CH_3COONa, 3H_2O$) in 300 ml water, add 2 ml glacial acetic acid and dilute to one litre.

4.1.2.8 *Standard zinc solution* (*0·05 M*) — Dissolve 3·26 g of electrolytic zinc granules (99·95 percent purity) in dilute hydrochloric acid (1:1) and evaporate the soultion to 1 to 2 ml, dilute with water add 50 ml buffer solution (*p*H 5·3), mix, make up, the volume to one litre. Standardize against standard aluminium solution following the method for determination of aluminium.

4.1.2.9 *Standard aluminium solution* (*0·05 M*) — Weigh accurately 1·349 0 g of pure aluminium wire (99·9 percent) in a 500-ml beaker containing 50 ml of water and 50 ml concentrated hydrochloric acid. Heat the solution to dissolve the metal. Cool and dilute the solution to 1 000 ml in a volumetric flask. (1 ml = 0·002 550 g Al_2O_3).

4.1.2.10 *Xylenol orange solution* — 0·5 percent (*m/v*) in water acidified with 2 drops of dilute hydrochloric acid (40 percent).

4.1.2.11 *Ammonium fluoride* — solid.

4.1.3 *Procedure*

4.1.3.1 Pipette out 50 ml aliquot from the main solution [*see* **5.3.3** of IS : 2000 (Part 2)-1985*] into 400-ml beaker and neutralize the solution with caustic soda solution. Dissolve the precipitate formed in minimum quantity of concentrated hydrochloric acid and pour the solution slowly with constant stirring to a 200-ml volumetric flask containing 25 ml of caustic soda solution and 1 g sodium carbonate. Keep this on the water-bath for about an hour and then cool to room temperature. Make up the volume to the mark with water and shake thoroughly. Filter dry and collect the filtrate in a dry beaker or dry polythene bottle.

4.1.3.2 Pipette out an aliquot (100 ml) from the filtrate to a 250-ml conical flask. Add about 30 ml of 0·05 M EDTA solution and a drop of methyl red indicator. Neutralize the solution with dilute hydrochloric acid, add 15 ml buffer solution and boil the solution for about 2 minutes. Cool to room temperature and titrate with standard zinc solution using 2 to 3 drops of xylenol orange indicator. The colour changes from yellow to red. Add about 2 g of ammonium fluoride to the solution and boil the solution for about 2 minutes. Add 10 ml buffer solution cool to

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*Methods of chemical analysis of bauxite: Part 2 Determination of silica (*first revision*).

the room temperature and then titrate with standard zinc solution adding a drop of xylenol orange indicator. The end point is from yellow to pinkish red. The percentage of alumina is calculated from the titre value of second titration with zinc.

4.1.4 *Calculation*

$$\text{Alumina (} Al_2O_3 \text{), percent} = \frac{A \times B}{C} \times 100$$

where

A = ml of zinc solution required for second titration,
B = equivalent Al_2O_3 (in g) per ml of zinc solution, and
C = mass in g of the sample as represented by the aliquot taken.

NOTE — This method is applicable when CaO and MgO is $< 0{\cdot}3$ percent (*m/m*).

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